

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/EP04/014382

International filing date: 15 December 2004 (15.12.2004)

Document type: Certified copy of priority document

Document details: Country/Office: IN

Number: 0047/MUM/2004

Filing date: 16 January 2004 (16.01.2004)

Date of receipt at the International Bureau: 04 February 2005 (04.02.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse

28.01.2005



INTELLECTUAL
PROPERTY INDIA
PATENTS / DESIGNS / TRADE MARKS /
GEOGRAPHICAL INDICATION



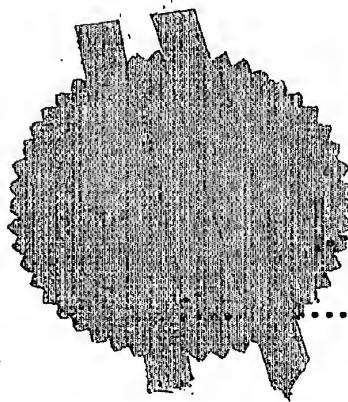
सत्यमेव जयते

Government Of India
Patent Office
Todi Estates, 3rd Floor,
Lower Parel (West)
Mumbai – 400 013

THE PATENTS ACT, 1970

IT IS HEREBY CERTIFIED THAT, the annex is a true copy of Application and Provisional Specification filed on 16/01/2004 in respect of Patent Application No.47/MUM/2004 of **HINDUSTAN LEVER LIMITED**, a company incorporated under the Indian Companies Act, 1913 and having its registered office at Hindustan Lever House, 165/166, Backbay Reclamation, Mumbai – 400 020, Maharashtra, India.

This certificate is issued under the powers vested in me under Section 147(1) of the Patents Act, 1970.



Dated this 18th day of January 2005.


(R. BHATTACHARYA)
ASST. CONTROLLER OF PATENTS & DESIGNS.

FORM 1

THE PATENTS ACT, 1970
(39 of 1970)

APPLICATION FOR GRANT OF A PATENT
[See Sections 5 (2), 7, 54 and 135 and rule 39]

1. We, HINDUSTAN LEVER LIMITED, a company incorporated under the Indian Companies Act, 1913 and having its registered office at Hindustan Lever House, 165/166, Backbay Reclamation, Mumbai - 400 020, Maharashtra, India

2. hereby declare -

(a) that we are in possession of an invention titled

IMPROVED PROCESS FOR TEA MANUFACTURE

(b) that the Provisional specification relating to this invention is filed with this application.

(c) that there is no lawful ground of objection to the grant of a patent to us.

3. further declare that the inventors for the said invention are

RAMASWAMY Seethalakshmi, 312, Priyam, 7th B Main, Koramangala, Bangalore - 560 034, Karnataka, India, Indian Citizen, NARAYANAN Venkatraj Venkatrao, 532, Ranka Court, 18 Cambridge Road, Bangalore 560 008, Karnataka, India, Indian Citizen and SHARMA Navin Kumar, A-109 Fem Saroj, 7 "A" Cross, LB Shastri Nagar, Vimanapura PO, Bangalore 560 017, Karnataka, India, Indian Citizen

4. We claim the priority from the applicant(s) filed in convention countries, particulars of which are as follows:-

5. We state that the said invention is an improvement in or modification of the invention, the particulars of which are as follows and of which we are the applicant/patentee.

6. We state that the application is divided out of our application, the particulars of which are given below and pray that this application deemed to have been filed on under Section 16 of the Act.

7. That we are the assignee of the true and first inventor

8. That our address for service in India is as follows:-

HV Williams & Co., Flats 1B and 1C "Monalisa", 17 Camac Street Calcutta - 700 017, India

47 | अक्टूबर | 2004
MUM | 17 | मुम | 2004

11 6 JAN 2004

16 | 1 | 04

16/1/04
6353
R.D.
S.D.M.

9. Following declaration was given by the inventor(s) :

We, the true and first inventors for this invention declare that the applicant herein is our assignee.

RAMASWAMY Seethalakshmi, 312, Priyam, 7th B Main, Koramangala, Bangalore – 560 034, Karnataka, India, Indian Citizen, NARAYANAN Venkatraj Venkatrao, 532, Ranka Court, 18 Cambridge Road, Bangalore 560 008, Karnataka, India, Indian Citizen and SHARMA Navin Kumar, A-109 Fem Saroj, 7 "A" Cross, LB Shastri Nagar, Vimanapura PO, Bangalore 560 017, Karnataka, India, Indian Citizen

RAMASWAMY Seethalakshmi
RAMASWAMY Seethalakshmi

NARAYANAN Venkatraj Venkatrao
NARAYANAN Venkatraj Venkatrao

SHARMA Navin Kumar
SHARMA Navin Kumar

10. That to the best of our knowledge, information and belief the fact and matters stated herein are correct and that there is no lawful ground of objection to the grant of patent to us on this application.

11. Followings are the attachments with the application:

- (a) Provisional Specification (2 copies)
- (b) Form-3
- (c) Fee Rs. 3000/- in Cheque

We request that a patent may be granted to us for the said invention.

Dated this 16th day of January 2004

To

The Controller of Patents
The Patent Office
Mumbai

HINDUSTAN LEVER LIMITED.

S Venkatramani
(S. Venkatramani)
Patents Manager

FORM -2

THE PATENTS ACT, 1970
(39 of 1970)

ORIGINAL

PROVISIONAL SPECIFICATION
(See Section 10)

IMPROVED PROCESS FOR TEA MANUFACTURE

HINDUSTAN LEVER LIMITED, a company incorporated under the Indian Companies Act, 1913 and having its registered office at Hindustan Lever House, 165/166, Backbay Reclamation, Mumbai -400 020, Maharashtra, India

The following specification particularly describes the nature of the invention and the manner in which it is to be performed.

47 | अंग्रेजी | 2004
MUM

16 JAN 2004

FIELD OF THE INVENTION

This invention relates to a process for obtaining cold water infusible or extractable tea starting with black tea as the raw material and to the products obtained thereby that have improved red colour, infuse faster and have good flavour.

BACKGROUND AND PRIOR ART

Leaf tea may be prepared as green leaf tea or black leaf tea. Generally, to prepare black leaf tea, fresh green leaves of the plant *Camellia sinensis* are withered (a process to allow the plucked tea leaves to lose moisture and bring about chemical / biochemical changes especially in aroma), macerated, fermented (in which process enzymes in the tea leaf use atmospheric oxygen to oxidise various substrates to produce coloured products) and then dried at higher temperatures (to stop the enzyme activities). Whereas, green tea is not exposed to the fermentation process and partial fermentation may be used to produce intermediate-type teas known as "oolong" tea.

Tea is consumed as a hot beverage or as a cold beverage (for example iced tea). The numerous compounds in the leaves that give the beverage its unique organoleptic properties are only sparingly soluble in cold water therefore tea is usually infused in water at temperatures close to 100 °C.

Cold water soluble teas are usually prepared by spray drying the liquor obtained by extraction of black tea or fibres generated during black tea manufacturing process. However, this process requires high temperatures or treatment with harsh chemicals like alkalis, which adversely affect the tea attributes like taste, colour and flavour.

There have been many methods reported to treat green tea to prepare tea products that infuse in cold water. US 4,051,264 (Lipton/Sanderson) describes a process that involves treating green tea leaves with enzymes during the processing of tea to generate cold water infusing teas. US 3,812,266 (Sanderson/Coggon) discloses a method that involves converting green tea to black tea using tannase and natural tea enzymes to convert green tea into black, and generate tea powders, which are both hot and cold water infusible.

Apart from the advantages of obtaining cold water infusible tea, an important consideration for the consumer is tea colour, brightness and aroma. Tea colour refers to the colour of the infusion with or without milk. Black tea infusions can range from yellow to red-brown in colour. Teas with bright, red liquor and good aroma are particularly preferred in countries like India and are perceived to be 'strong' teas. It is thus desirable to produce tea with these characteristics. Moreover, it is desirable to produce tea that infuses faster in water as these teas are perceived to be strong teas. Thus teas that infuse fast and provide a good red colour are much preferred by consumers and are perceived to be teas with good strength.

Many methods of treating green tea to produce tea products that give bright red colour liquor in water have been reported. US 5863581 (Lipton, Division of Conopco, Inc.) discloses a process for manufacturing a tea product where zeolites are used to generate red coloured teas. Tadao Kurata et al in Agr. Biol. Chem, 37 (6), 1471-1477, 1973 discloses that a red pigment is produced at the initial stage of the browning reaction of dehydro-L-ascorbic acid (DHA) with alpha-amino acid. 5-phenyl-3,4-diketo-gamma-butyrolactone, which has the same type of lactone ring structure as dehydro-L-ascorbic acid, is said to give a similar red colour when reacted with alpha-amino acid. The pigment is said to have the same structure as the red pigment that is produced by the oxidation of L-scorbamic acid.

WO 01/70038 (Unilever, 2001) concerns a process for manufacturing a cold water infusible black leaf tea comprising macerating freshly plucked tea leaves, allowing them to ferment, firing the leaves to arrest fermentation and then drying them to yield black leaf tea. The process is characterised in that the tea leaves are treated with a solubilising compound selected from the group consisting of ascorbic acid, dehydroascorbic acid, L-scorbamic acid or 5-phenyl-3,4-diketo-gamma-butyrolactone, preferably during the maceration step. The black leaf tea so produced is soluble in water at 5 to 100 °C. The teas have a good red colour. The publication also teaches that the tea leaves are preferably treated with the solubilizing compound in the presence of oxidative enzymes and/or hydrogen peroxide to enhance the tea flavour and colour.

JP 47-49719 (Tanabe, 1972) reports a method of improving the taste and flavour of tea during its manufacture by adding amino acids and 1,3-dihydroxy-2-propanone at an appropriate heating stage.

GB 2348 104 (Unilever, 2000) reports a method of manufacturing black tea with improved aroma comprising the step of treating green leaf tea with a combination of phenolic acids and amino acids prior to fermentation step.

JP 02128669 (Ajinomoto, 1990) reports a method to improve flavour and reduce bitter taste of tea by adding amino acid wherein amino acid and tea are in the range of 0.1 to 60% and 0.1 to 50% respectively.

All of the above methods have been reported to treat green tea during the process of manufacture of black tea to prepare cold water infusible tea or enhancing other attributes such as flavour & bitterness reduction. There has been a need to provide for cold water infusible teas that has good red colour and more importantly fast infusion rate in water starting with black tea as the raw material. This is especially useful in countries where there are no tea gardens and there are no tea processing factories. Black tea is a comparatively more stable product and so black tea can be imported and the process of making this fast infusing cold water infusible tea can be carried out at location far remote from the tea gardens. The present inventors have now found that one can prepare a tea product that provides a high quality tea beverage when infused in hot or cold water and provides a rich bright red colour infusion in fast time by treating black tea with ascorbic acid or its salts/derivatives, one or more amino acids and an oxidising agent.

Objects of the Invention

It is thus an object of the invention to provide for a cold or hot water infusible tea product with improved red colour and good flavour.

It is a further object of the invention is to provide for a cold or hot water infusible tea that infuses quickly into the water.

Definition of the Invention

According to the first aspect of the invention, there is provided a process for manufacturing a tea product comprising contacting black tea with ascorbic acid or its salts/derivatives, one or more amino acids as herein described, an oxidizing agent and water for a period of at least 5 minutes followed by drying to prepare a tea product that is infusible in water at 5 to 100 °C.

According to a preferred aspect of the invention, there is provided a process for manufacturing a black tea product comprising contacting black tea with aqueous solutions of, by weight of black tea

1 to 8% ascorbic acid or its salts/derivatives,

0.1 to 8% of one or more amino acids as herein described and

0.3 to 7% of an oxidizing agent

for a period of at least 5 minutes followed by drying to less than 5% moisture to prepare a black tea that is infusible in water at 5 to 100 °C.

According to a further preferred aspect of the invention, there is provided a process for manufacturing a tea product comprising of contacting black tea with aqueous solutions of, by weight of black tea

2 to 4% ascorbic acid or its salts/derivatives

0.1 to 2% of one or more amino acids as herein described and

0.3 to 5% of hydrogen peroxide

for a period of 5 minutes to 24 hours followed by drying to less than 5% moisture to prepare a tea product that is infusible in water at 5 to 100 °C.

The invention also pertains to the tea obtained by this process. The tea so obtained is cold water and hot water infusible/extractable and the infusion/extract shows improved red colour. The tea also infuses quickly into the water.

It is particularly preferred that the black tea is first contacted with the ascorbic acid or its salts/derivatives before it is contacted with the oxidising agent.

Detailed Description of the Invention:

"Tea" for the purposes of the present invention means leaf material from *Camellia sinensis* var. *sinensis* or *Camellia sinensis* var. *assamica*. It also includes rooibos tea obtained from *Aspalathus linearis* however that is a poor source tea. "Tea" is also intended to include the product of blending two or more of any of these teas.

"Leaf tea" for the purposes of this invention means a tea product that contains one or more tea origins in an uninfused form.

"Cold water infusible" for the purposes of this invention means giving good colour, flavour and mouthfeel in a short infusion time i.e. less than 10 minutes, but preferably less than 5 minutes at a temperature at or above 5 °C.

For the avoidance of doubt the word "comprising" is intended to mean including but not necessarily "consisting of" or "composed of". In other words the listed steps or options need not be exhaustive.

Tea manufacture, especially black tea manufacture, traditionally comprises: withering, macerating, fermenting and firing. Black tea for the purpose of the invention is obtained by the above process or by any other known process to prepare black tea.

The present invention concerns further treatment of the black tea obtained as described above to prepare a cold water infusible tea product. The treatment involves contacting black tea (leaf /dust) or secondaries or green leaf mixed black tea or off graded black tea or tea solids coated black tea with ascorbic acid or its salts/derivatives, one or more amino acids, and an oxidising agent preferably hydrogen peroxide with or without peroxidase addition, in the presence of moisture, in order to enhance the infusion of the black tea in cold water. All these agents are preferably added in the form of aqueous solutions. Alternately they may be dry mixed followed by addition of water. The tea is thus contacted for atleast 5 minutes and preferably dried to less than 5% moisture. The solution can be applied singly or in split doses. The treatment is preferably given in the form of a spray or dip.

The black tea can be treated either ex-drier mouth or after grading. Ascorbic acid or its salts/derivatives including sodium or calcium salts are preferably added at a concentration ranging from 1% to 8% by weight of tea, more preferably at a concentration ranging from 2% to 4% by weight of tea. The ascorbic acid or its salts are preferably added as an aqueous solution.

Amino acid can be used singly or in combination. Amino acids as per this invention include, alanine, arginine, asparagine, cystine, glutamine, glycine, histidine, isoleucine, leucine, lysine, methionine, phenylalanine, serine, threonine, tryptophan, tyrosine, valine. They are either mixed with ascorbic acid or can be sprayed separately and its concentration is between 0.1% to 8% by weight of tea, more preferably at a concentration of 0.1% to 2% by weight of tea. The resultant solution of ascorbic acid and amino acid can be applied singly or in split doses. The treatment is preferably given in the form of a spray or dip.

Preferably, aqueous solutions of ascorbic acid or its salts/derivatives and amino acids should be well heated before contacting with tea. The amount of water can be between 10% to 100% by weight of tea, more preferably at a concentration of 30% to 100% by weight of tea, most preferably at a concentration of 40% to 100%.

It is preferred that the oxidising agent is added to the black tea after the ascorbic acids and its salts/derivatives and amino acid is added. Oxidising agent may be Hydrogen peroxide, Calcium peroxide, Magnesium peroxide, oxygen itself or any compound, which can release oxygen under the process conditions, or could be an enzyme which can release hydrogen peroxide. Preferred oxidising agent is hydrogen peroxide. The oxidising agent is preferably added at a concentration of 0.3% to 7% by weight of tea. When the oxidising agent is hydrogen peroxide it may be added as a 10% to 75% solution in water. Usually hydrogen peroxide is available at 30% hydrogen peroxide solution in water. The oxidising agent is more preferably added at a concentration of from 0.3% to 5% by weight of tea. The solution can be applied singly or in split doses. The treatment is preferably given in the form of a spray or dip.

The tea is preferably kept contacted with black tea, a process step known as incubation for a period of atleast 5 minutes. The incubation may be carried out for a period of up to 24 hours. The incubation is preferably carried out at a temperature of 10 to 60 °C.

The tea can be dried using any method/process known in the art, preferably giving tea with a moisture content of less than 5%.

Water infusions of the tea produced by the process described above in water at temperatures in the range of 5 to 100 °C will give tea with superior colour and flavour.

Any low liquor or off-grade tea can be converted to value attributing component by way of this invention without any wastage.

The process of the invention shall now be described with reference to the following non-limiting examples:

EXAMPLES

Comparative Example A:

100 grams of black tea was wetted with 100mL hot water. This was incubated around 25 - 30°C for about 15 minutes followed by drying on a fluidised bed drier at 130 - 140°C to bring down the moisture to less than 5% on black tea basis.

Comparative Example B:

A process as per Comparative Example -A was carried out except that additionally 5 ml of 30% hydrogen peroxide was sprayed immediately after the addition of water.

Comparative Example C :

A process as per Comparative Example -A was carried out except that a solution of 0.5 g of phenyl alanine in 100 mL water, which had been heated on a boiling water bath for 5 minutes, was sprayed on the tea instead of the 100 mL of water used in the comparative example - A..

Comparative Example D :

A process as per Comparative Example -C was carried out except that 3g of ascorbic acid was used in place of 0.5 g of phenyl alanine.

Comparative Example E :

Ascorbic acid (3 g) was dissolved in 100 mL of hot water. This solution was further heated on a boiling water bath for 5 minutes. The hot solution was sprayed on 100 grams of black tea. About 5 mL of 30% hydrogen peroxide solution was added to it. This was incubated at 25 - 30°C for about 15 minutes followed by drying on a fluidised bed drier at 130 - 140°C to bring down the moisture to less than 5% on black tea basis.

Comparative Example F :

A process as per comparative example – E was carried out except that 0.5 g of phenyl alanine was used instead of 3 g of ascorbic acid.

Comparative Example G :

Ascorbic acid(3 g) and phenyl alanine (0.5 g) were dissolved in 100 mL hot water. This solution was heated on a boiling water bath for 5 min: The hot solution was sprayed on to 100 grams of black tea. This was incubated at 25 - 30°C for about 15 minutes followed by drying on a fluidised bed drier at 130 - 140°C to bring down the moisture to less than 5% on black tea basis.

Example H :

A process as per comparative example – E was carried out and additionally 0.5 g phenyl alanine was added to the 100 ml water.

The tea thus prepared in Comparative examples A to G and Example H were tested for infusion in water by the following method:

Process for preparing cold water infusions

1g of black tea samples were Infused in 100 ml of water at 25°C for 5 minutes. The solution was filtered to give cold tea infusions and the colour of infusions obtained from Comparative Examples A-G and Example H was measured using a Hunter Lab Ultrascan XE™ colorimeter in the transmittance mode. The data is presented in Table 1.

Colour measurements

L*a*b* measurements for colour were carried out on Hunterlab UltraScan XE™ colorimeter under the following conditions:

Cuvette 2cms (Quartz), Mode Transmittance, Illuminant D65, Observer 10, Scale CIELAB. The reflectance at 520 nm was also determined.

50 ml of the brew was taken in a 2 cm quartz cuvette, transmittance/reflectance was measured under the conditions mentioned above.

The a* values are reported as these denote redness of the tea. The higher the value, the redder the tea. 520 nm is the wavelength at which the red colour absorbs. The higher the absorbance value the greater the red colour.

Table 1:

| Example | a* | Absorbance 520 nm |
|---------|------|----------------------|
| A | 28.6 | 0.77 |
| B | 30.7 | 0.85 |
| C | 21.4 | 0.59 |
| D | 28.3 | 0.78 |
| E | 41.5 | 1.10 |
| F | 29.4 | 0.83 |
| G | 25.0 | 0.66 |
| H | 50.4 | 1.51 |

Data in Table-1 indicates that there is synergistic benefit in adding ascorbic acid, phenyl alanine and hydrogen peroxide to the black tea compared to adding any one or combinations of any two of these ingredients.

Rate of Infusion

The rate at which the tea infuses into cold water was also studied. The effect was studied for Comparative Examples A, E & Example H. The colour of the infusion after 30 seconds, 3 and 5 minutes was determined. The data is presented in Table 2.

Table 2:

| Example | Time (minutes) | A* | Absorbance 520nm |
|---------|----------------|-------|---------------------|
| A | 0.5 | 4.31 | 0.28 |
| | 3 | 18.91 | 0.55 |
| | 5 | 23.98 | 0.66 |
| E | 0.5 | 26.71 | 0.64 |
| | 3 | 30.27 | 0.73 |
| | 5 | 39.06 | 1.02 |
| H | 0.5 | 31.98 | 0.72 |
| | 3 | 45.24 | 1.17 |
| | 5 | 49.93 | 1.49 |

The data presented in table 2 shows that the teas prepared by the process of the invention (Example-H) infuse faster and give better red colour within a short period as compared to control teas or teas treated with ascorbic acid and hydrogen peroxide alone.

It is thus possible by way of the present invention to produce a black leaf tea that infuses quickly in hot or cold water and provides for a good red colour.

Aroma/Flavour enhancement – Head Space Gas Chromatography:

The additional benefit of this invention which is aroma / flavour enhancement is demonstrated by measuring the levels of key pleasant flavoured molecules like Phenyl acetaldehyde and benzaldehyde. The data for the comparative examples from A to G and example H are given in Table 3.

Table 3:

| Example | Phenyl acetaldehyde (Peak area) | Benzaldehyde (Peak area) |
|---------|---------------------------------------|-----------------------------|
| A | 15349 | 33937 |
| B | 11063 | 28654 |

| | | |
|---|--------|-------|
| C | 9683 | 42449 |
| D | 8522 | 37137 |
| E | 6679 | 50156 |
| F | 153824 | 77466 |
| G | 75130 | 92055 |
| H | 177034 | 96077 |

Comparitive Example-J

An experiment as per Example-H was carried out without using black tea and the colour a^* was measured at zero, 30 and 60 minutes in comparison to Example H and Comparitive Example-A. The data is summarized in Table-4

Table 4:

| Example | a^* at Zero time | a^* after 30 minutes | a^* after 60 minutes |
|---------|--------------------|------------------------|------------------------|
| A | 21.79 | 17.17 | 15.84 |
| H | 50.63 | 54.27 | 53.65 |
| J | -0.38 | -0.62 | -0.79 |

Table 4 demonstrates that black tea is essential for producing the red colour.

It is thus possible by way of the present invention to produce a black leaf tea that infuses quickly in hot or cold water and provides for a good red colour.

Dated this 16th day of January 2004

HINDUSTAN LEVER LIMITED

S Venkatramani
(S. Venkatramani)

Patents Manager

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/EP04/014382

International filing date: 15 December 2004 (15.12.2004)

Document type: Certified copy of priority document

Document details: Country/Office: GB

Number: 0408250.9

Filing date: 14 April 2004 (14.04.2004)

Date of receipt at the International Bureau: 04 February 2005 (04.02.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse